

In re the application of:

SAKASHITA et al

Group Art Unit: 1713

Serial Number: 09/530,202

Examiner: Dr. Kelechi C. Egwim

Filed: April 26, 2000

For: PROCESSING AID FOR VINYL CHLORIDE RESIN AND VINYL

CHLORIDE RESIN COMPOSITION

### **DECLARATION UNDER 37 CFR 1.132**

Honorable Commissioner Washington, D.C. 20231

Sir,

Yasushi Nakanishi residing at 4-1-13, Takasago-cho Okihamamachi, Takasago City, Hyogo, JAPAN duly deposes and says:

- 1. That graduated from he Department Macromolecular Science, Faculty of Science, Osaka University, Osaka, Japan, in the year 1990, and he received the degree of Master of Macromolecular Science from Osaka University, Osaka, Japan, in the year 1992;
- 2. That since 1992, he has been employed in the capacity of KANEKA CORPORATION;
- 3. That from 1992, he has been engaged in research and development for impact modifiers and processing aids of plastics;

- 4. That he has read and is familiar with the instant application for United States Letters Patent and Office Action thereto mailed November 14, 2002; and
- 5. That he has made experiments in order to prove that a polymer obtained by a process described in the cited reference have low specific viscosity ( $\eta_{sp}$ ), and the obtained polymer does not sufficiently work as a processing aid for poly(vinyl chloride).

#### EXPERIMENT

Specific viscosity, transparency (all light transmittance and claud value), gelation property and foamability were estimated as mentioned below.

[Measurement of specific viscosity ( $\eta_{sp}$ )]

Into 100ml of chloroform, 0.1g of a sample was dissolved and viscosity was measured by employing Ubbelohde's viscometer maintained at a constant temperature in 30°C water bath.

# [Gelation property]

To 100 parts by weight of poly(vinyl chloride) (average molecular weight 680) were added 6.0 parts of a processing aid, 5.0 parts of calcium carbonate, 3.0 parts of titan oxide, 3.0 parts of dibasic lead phosphite, 0.4 part of lead stearate, 0.8 part of calcium stearate, 0.3 part of hydroxystearic acid (LOXIOL G-21 available from Henkel company), an aliphatic carboxylate ester (LOXIOL G-32 available from Henkel company), 0.4 part of dibasic ester of an aliphatic alcohol (LOXIOL G-60 available from

Henkel company) and 0.5 part of oxidized polyethylene wax (Hoechst-Wachs PED-521 available from Hoechst), and then they were mixed by means of a Henschel mixer. After the inner temperature was raised to  $110^{\circ}$ C, the mixture was cooled to obtain a powder compound.

Gelation property was evaluated by a slope of a straight line between a maximum torque and a minimum torque on kneading time vs. torque curve, which was obtained by kneading 62g of the obtained powder compound at 150℃ by means of a small kneading machine (Plasticoder PLE-331 made by Brabender). It was judged that the gelation property was better in case of a bigger slope.

## [Transparency]

To 100 parts of poly(vinyl chloride) (average molecular weight 680) were added 3.0 parts of a processing aid, 1.5 parts of an octyl tin mercaptide stabilizer, 1.5 parts of an epoxidized soybean oil, 1.0 part of butyl steatate and 0.5 part of a polyethylene glycol fatty acid ester, and then they were mixed by means of a Henschel mixer. After the inner temperature was raised to  $110^{\circ}$ C, the mixture was cooled to obtain a powder compound. The vinyl chloride resin composition was milled at  $170^{\circ}$ C for five minutes by means of 8 inches test roll, then pressed at  $180^{\circ}$ C for fifteen minutes to prepare a pressed plate having 3mm thickness. All light transmittance and cloud value of the obtained pressed plate were measured according to JIS-6714. It is indicated that the transparency is more excellent in case of the bigger all light transmittance. And it is also

indicated that the transparency is more excellent in case of the smaller cloud value.

[Foamability]

To the compound used in the evaluation of gelation property was added 1.0 part of azo dicarboxylamide to obtain a powder compound in the same manner as the above-mentioned method. And then the compound was extruded at 170°C by a small conical screw extruder (2D20C) attached to a labo plastmill (made by Toyoseiki company) in order to produce a foamed profile. Specific gravity of the obtained profile was measured. It is indicated that the expansion ratio is bigger and the formability is more excellent in case of the smaller specific gravity.

EXPERIMENT 1 (USP 5,093,420 Comparative Example 5) (EP392465 page 5 line 52 to page 6 line 4)

This procedure is the same as that in Comparative Example 5 of USP 5,093,420.

A reactor equipped with a stirrer was charged with 200 parts of water, 1 part of dioctyl sodium sulfosuccinate and 0.01 part of potassium persulfate, and oxygen is removed from the space of the reactor and water by introducing nitrogen into the reactor. The mixture was raised to a temperature of 65°C with stirring, to which 80 parts of methyl methacrylate was added over 4 hours, and the polymerization reaction was continued for 1 hour with stirring while heating to substantially complete the polymerization reaction (first step). Then, to the reaction

mixture was added a monomer mixture of 11 parts of butyl acrylate and 9 parts of methyl methacrylate over 1 hour, and the temperature of the mixture was kept at 65°C for 1.5 hours, then the temperature was cooled down to 40°C (second step).

Concerning a polymer obtained by this procedure, specific viscosity was estimated by the above-mentioned method. Specific viscosity of the first-step polymer and the second-step polymer were 0.64 and 0.58, respectively.

Furthermore, the obtained polymer was added to poly(vinyl chloride) to obtain a poly(vinyl chloride) composition and the composition was estimated as to gelation property, transparency (all light transmittance and cloud value) and foamability by the above-mentioned methods. In result, all light transmittance was 53 %, cloud value was 22 %, gelation property was 4.5 Nm/min. and formability was 0.70 g/cm<sup>3</sup>.

### CONCLUSION

The second-step polymer obtained by the procedure described in USP 5,093,420 has specific viscosity of above 0.5, but specific viscosity of the first-step polymer is less than 0.7.

Furthermore, as is clear from results, a poly(vinyl chloride) composition obtained by adding the polymer as a processing aid to poly(vinyl chloride) does not have satisfactory gelation property, transparency and foamability.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

> day of March, 2003 This 6th

Yasushi Nakanishi

We, the undersigned witnesses, hereby acknowledge that Yasushi Nakanishi is personally known to us and did execute the foregoing Declaration in our presence on:

Date: March 6, 2003

Witness Hiroshi Fone

Date: March 6, 2003